





The Patent Office Cardiff Road Newport

Newport Gwefit\_\_

2 2 JUL 1998

**WIPO** 

NP9 IREC'D

PCT

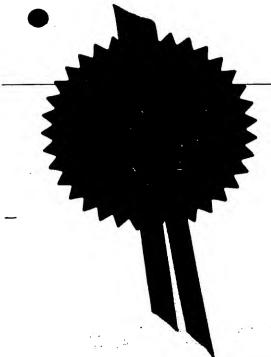
09/424052

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation and Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



Signed

Dated

07 JUL 1998

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

An Executive Agency of the Department of Trade and Industry

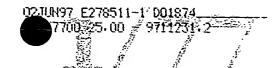
applicant, or

See note (d))

c) any named applicant is a corporate body.

ents Act 1977 (kule 16)









The Patent Office

Cardiff Road Newport Gwent NP9 1RH

|                |   | CSIVED BY P   | Gwent NP9 1RH                          |
|----------------|---|---|--|
| 1.             | Your reference  | CG/97057GB  |  |
| 2.             | Patent application number (The Patent Office will fill in this part)  | 971   | 1231.2                                 |
| 3.             | Full name, address and postcode of the or of each applicant (underline all surnames)  | Kemin Industries, Inc<br>2100 Maury Street,<br>Box 70, Des Moines,<br>IA 50301, |  |
|                | Patents ADP number (if you know it)   | u.s.a. 0722   | 2191400                                |
|                | If the applicant is a corporate body, give the country/state of its incorporation   | United States of Amer   | ica                                    |
| 4.             | Title of the invention  |   |  |
|                |   | Food Processing Proced  | lure                                   |
| <br>5.         | Name of your agent (if you have one)  | Batchellor, Kirk & Co   |  |
|                | "Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)   | 2, Pear Tree Court,<br>Farringdon Road,<br>LONDON EC1R ODS                      |  |
|                | Patents ADP number (if you know it)   | 0000315001  |  |
| 6.             | If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these  | Country Priority application number (if you know it)                            | Date of filing<br>(day / month / year) |
|                | earlier applications and (if you know it) the or each application number  |   |  |
| <del></del> 7. | If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application  | Number of earlier application   | Date of filing<br>(day / month / year) |
| <br>8.         | Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:  a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an | Yes   |  |

## Patents Form 1/77

 Enter the number of sheets for any of the following items you are filing with this form.
 Do not count copies of the same document

| Continuation sheets of this form                                   |   |       |
|--|---|-------|
| ,  |   | 4     |
| Description  | 7   | •     |
| Classes  |   |       |
| Claim(s)   |   |       |
| Abstract   | . <b>-</b>  |       |
|  |   |       |
| Drawing(s)   | -   |       |
| 10. If you are also filing any of the following,                   |   |       |
| state how many against each item.                                  |   |       |
| Priority documents   | _   |       |
|  |   |       |
| Translations of priority documents                                 | -   |       |
| Statement of inventorship and right                                | <u>-</u>  |       |
| to grant of a patent (Patents Form 7/77)                           |   |       |
|  | •   |       |
| Request for preliminary examination and search (Patents Form 9/77) | -   |       |
| and scarcii (ratents rorm 9///)                                    |   |       |
| Request for substantive examination                                |   |       |
| (Patents Form 10/77)   | - ·   |       |
|  |   |       |
| Any other documents  | -   |       |
| (please specify)   |   |       |
| 11.  | I/We request the grant of a patent on the basis of this applica | tion. |
|  |   |       |
|  | Signature Date  |       |

Name and daytime telephone number of person to contact in the United Kingdom Batchellor, Kirk & Co.
Carol GREAVES

Tel: 0171-253 1563

## Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

## **Notes**

- a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- b) Write your answers in capital letters using black ink or you may type them.
- c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- e) Once you have filled in the form you must remember to sign and date it.
- f) For details of the fee and ways to pay please contact the Patent Office.

7th May 1997

## Food Processing Procedure

The present invention relates to the preparation of food additives, in particular to the preparation of flavours for incorporation into foodstuffs, as well as to the additives and foodstuffs, particularly animal feeds, obtained thereby.

10 Many foodstuffs for both human and animal consumption are processed for the incorporation of additives. With respect to animal feeds, a clear relationship between food flavour and feed intake has been recognised.

Hitherto, flavour compounds have been incorporated into 15 foodstuffs, particularly animal feeds, by addition to carrier particles, in particular, salt particles. flavour compounds in liquid or powder form are distributed onto fine salt particles which may then be added to foodstuffs. The particle size of the salt 20 particles used as a carrier is necessarily small in order to ensure that the carrier presents a large as possible surface area onto which the flavour may be adsorbed so as to optimise the amount of flavour to the amount of carrier. The problem here is that the final product is 25 dusty, being only of a similar size to the fine salt particles utilised as the carrier. Significant quantities of dust in the atmosphere is undesirable, particularly in industrial environments since small

30 particles present in the air, may be breathed in by workers in the area, and may adversely affect health.

Furthermore, being merely coated on the surface of the carrier particles, the flavour compounds may be

35 relatively easily and rapidly lost, for example, by abrasion and/or vapourisation leading to a reduced shelf life or persistance of flavour of both the additive and the finished product (foodstuff).

The applicants have found a improved method of formulating food additives which produces a more easily handled and robust product.

5

According to the present invention, there is provided a method of preparing a food additive, said method comprising

- (i) mixing crystalline carrier particles with an10 additive compound;
  - (ii) applying to the thus produced mixture a solution of a crystalline solid under conditions such that the said carrier particles are partially dissolved; and
- (iii) allowing the mixture to cocrystallize and to aglomerate so as to form particles of increased average size as compared to the said carrier particles.

During the cocrystallization step (iii), the particles

20 grow and/or coalesce. Thus the average particle size of
the product is generally larger than the particle size of
the carrier molecules themselves. Such particles will
present a reduced dust hazard as compared to the carrier
particles themselves.

25

Furthermore, during the cocrystallisation in step (iii), the additive becomes incorporated into pores in the resultant cyrstal structure. The crystals are, by and large, coated with the crystalline solid, so that the

additive is entrapped within the larger particles. This means that the additive is not easily lost or degraded by other components in the foodstuff. Therefore, the shelf life of the food additive, as well as the feed or food product incorporating the additive is also increased.

35

Suitable carrier particles are non-toxic crystalline solids such as salt or sugar crystals. In particular alkali metal halides in particular chlorides such as

sodium or potassium chlorides are preferred carrier particles. Such compounds are economical and readily available and are generally permitted in animal feeds.

The carrier particles may comprise a mixture of the carriers and also dessicating or drying agents as `cocarriers'. These agents include edible silica and silicates, for example Sipernate® 22 available from Degussa. These agents can absorb up to 2.5 times their own weight of moisture. Inclusion of such agents may be 10 preferable where the additive compound is in the form of a liquid (either a liquid per se or a solution such as an aqueous solution), likely to form a `sticky' product when combined with the carrier. The amount of such a cocarrier required will depend upon the amount of additive compound as well as the precise nature of the additive compound and the carrier. In general, the weight ratio of co-carrier to liquid additive compound would be in the region of about 1:2 so that if 20 g of liquid additive were to be used, the carrier would include 10g of cocarrier.

15

20

Preferably the carrier particles have have an average particle size of from 50-300 microns, and generally in the region of about 200 microns. 25

Examples of food additives which may be incorporated in this way include flavours, vitamins in particular vitamin E or vitamin A or other diet supplements, preservatives,

colorants, etc. The additives are suitably in the form 30 of liquids or solids at room temperature. The method is particularly suitable for the inclusion of flavours for example natural flavours such as vanilla or flavour composites containing such as Butter Vanil Arome Dry® (available from Kemin Europa N.V.), as well as artificial 35 flavours such as pyrazines, esters and ketone flavours as would be understood in the art.

The ratio of the food additive to carrier particle which are mixed together in step (i) depends upon the precise nature of the carrier particles and the additive compounds employed. However, a weight ratio of carrier (including co-carrier where present) to additive of from 1:1 to 99:1, suitably about 4:1 will generally be acceptable.

The solution utilised in step (ii) of the process of the
invention is suitably an aqueous solution of a material
which forms a non-toxic crystalline solid at room
temperature. The solid preferably has a palatable
flavour of its own, so that it may enhance the flavour of
the additive. Examples of such compounds are sugars,
such as glucose, maltose or fructose or other sweeteners.
Such compounds are readily available and low in cost
which is particularly important for animal feed
applications.

The solution of crytalline solid used in step (ii) is suitably a concentrated aqueous solution, for example of from 30-65%, preferably about 40% crystalline solid. This is particularly true for sugar solutions where concentrations in excess of 65% are regarded as being syrups.

The solution must be applied in such a way that it results in only partial dissolution of the carrier particles. The conditions under which this will happen

depends upon the precise nature of the carrier particles as well as the nature of the solution of crystalline solid. A preferred application method is by spraying the solution onto the particles, and thus it is preferable that the solution is able to pass through a nozzle of a spray applicator, most preferably a fine nozzle of from 0.46mm to 0.97mm in size. A flow rate of from 0.28 1/min to 0.84 1/minute, suitably at about 0.56 1/minute may be utilised.

When applied in this way, the spray droplets of the water/sugar solution wet the contact surface of the salt containing the additive, such as flavour, components. As a result, co-crystallisation of the sugar and the outer layer of the salt occurs, together with coating of the contact area with the liquid sugar solution. The additive, such as the flavour, becomes stably incorporated into the particles thus formed.

10

The solution may optionally include other active components such as flavour enhancers for example maltol or heliotropine, artificial sweeteners such as saccharine, or fixatives.

15

20

Conditions under which the solution of crystalline solid is applied in step (ii) will vary depending upon the precise nature of the carrier molecules, the additive, the crystalline solid itself as well as the process equipment employed. However temperatures of from 10°C to 50°C, suitably at about 25°C may be used.

The amount of solution applied to the carrier/additive mixture to achieve the desired product will once again depend upon the precise nature of the components and the process equipment. However, in general, the amount of solution applied will be of the order of from 5 to 25wt% of the amount of the additive in the mixture, suitably at about 10% by weight.

30

35

The cocrystallisation and aglomeration of step (iii) of the process of the invention may occur or begin to occur during the spraying step (ii) depending upon the particular conditions employed. After spraying, the sprayed product is subjected to conditions which would allow co-crystallisation and aglomeration of the components to occur or to continue, as necessary. This may include maintaining the product at moderate

temperatures for example of from 15° to 50°C, optionally in a moving air stream for example as may be found in a fluidised dryer.

Once prepared in this way, the product is generally quite dry but optionally may be subjected to extra drying. This can be accomplished using a fluid bed dryer with an air temperature of from about 60 to 150°C, suitably at about 100°C and a product temperature of about 25°C.

Drying times may be relatively short, for example from 2 to 20 minutes.

The resultant particles generally have an particle size in excess of 300 microns, for example about 500 microns.

15

Thus the invention further provides a particulate food additive comprising a non-toxic soluble salt, a food additive and a sugar which have been subjected to co-crystallization.

20

25

Such food additive particles can be admixed with foodstuffs and in particular animal feeds in a conventional manner. The size of the particles means that the handling process can be carried out more easily and with less dust than hitherto.

In yet a further aspect, there is provided a foodstuff comprising a particulate food additive as described above.

30

Another aspect of the invention provides a method for supplementing a foodstuff, said method comprising mixing said foodstuff with a particulate food additive in the form of a particulate material as described above.

35

The invention will now be particularly described by way of example.

Example 1

15

Butter Vanille Arome Dry flavour ®(250g) from Kemin Europa N.V. contains vanilla on a carrier comprising sodium chloride (74wt%) with a silicate as a co-carrier (10wt%). This product has an average particle size of 0.2mm.

A sugar solution (300g sugar in 750ml water) was 10 prepared. 26 g of the sugar solution was applied to the Butter Vanille Arome Dry® by spraying.

The resultant crystalline product was transferred to a fluid bed dryer and dried with an air temperature of 100°C and a product temperature of 30°C for five minutes.

The resultant product was in the form of uniform rounded granules having an average particle size of 0.5mm.

. . .

PCT GB98/01549

Batchellor, Link 160.

20/6/08.